

ZEISS ULTRA-60 FIELD EMISSION SCANNING ELECTRON MICROSCOPE (FE-SEM) PROCEDURE

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SPECIAL NOTES OR RESTRICTIONS:

- Must be qualified to use the tool by super-user.
- Must be given a Smart-SEM account by super-user.
- Always use the specimen exchange assembly to load and unload samples. If a sample breaks or becomes stuck in the specimen chamber, notify super-user or tech support. **DO NOT ATTEMPT TO REMOVE.**
- If the red OFF light or the yellow STANDBY light is illuminated, contact super-user or tech support. **DO NOT ATTEMPT TO START THE INSTRUMENT.**
- **ALWAYS TURN ON THE CHAMBER CAMERA BEFORE ATTEMPTING TO RAISE THE STAGE.** Failure to observe the interior of the specimen chamber may result in damage to the SEM and the sample. Never adjust x, y, rotate, or tilt if there is a chance that the sample may be touching the lens cap.
- When screwing the sample exchange rod into the sample holder, do not overtighten.

SAFETY PRECAUTIONS:

- **This instrument may generate radiation during operation.** It is strictly prohibited to remove any cover panels, particularly those on the electro-optic column and the specimen chamber.
- The acceleration voltage is not permitted to exceed 30 kV.
- Keep the area in front of all ventilation openings clear to prevent fire hazard and overheating of electronics.
- Do not bump into the specimen exchange assembly or apply pressure that may bend the specimen exchange rod.

SAMPLE LOADING:

- Check the FE-SEM vacuum/electronics status panel. If **GREEN**, OK to proceed. If **YELLOW** or **RED**, stop immediately and contact super-user or tech support.
- Verify that EM server is running on the right hand LCD. If not, contact super-user or tech support.
- Click on the Smart-SEM icon on the bottom of the left hand LCD. Enter user name and password to log on and activate the SEM interface.
- Choose sample holder and mount sample with carbon tape, paint, or clips (if applicable). Sample holders are available for 4-inch wafers, wafer pieces, cross-sections, and mounting samples at an angle. The small orange-handled screwdriver is used to mount the pin-type mounts in the multi-angle holder.
- Press the **CAMERA** button on the keyboard. The interior of the specimen chamber will appear.
- Press the **EXCHANGE** button on the keyboard. A dialog box will pop up. When it says "Ready to Exchange" position yourself in front of the sample exchange assembly and wait for the green **PROCEED** light to illuminate.
- Check that **CLOSE** and **PUMP** buttons are both illuminated and **OPEN** button is dark. This indicates that the sample exchange area is under vacuum and the door separating the specimen chamber from the sample exchange chamber is closed. You can verify that the door is closed by looking through the sample exchange window.
- Press **PUMP** button (light will go out).
- Press **PURGE** button (light will illuminate). You will hear nitrogen flow into the sample exchange chamber and the door will be released.
- Pull the door back and hook the door latch to hold it open.
- Slide the sample holder onto one of the two mounts and gently screw the sample exchange rod into the sample holder. Do not overtighten.
- Release the door latch and gently push the door closed.
- Press **PURGE** button (light will go out).
- Press **PUMP** button (light will illuminate) and the sample exchange chamber will pump down.

- *** Wait for the green **PROCEED** light to illuminate. This means the sample exchange chamber is pumped down.
- Press **CLOSE** button (light will go out).
- Press **OPEN** button (light will illuminate). The door between the sample exchange chamber and the specimen chamber will open.
- Gently release the specimen rod latch.
- Carefully, without applying any bending pressure to the rod, slide the sample exchange rod into the specimen chamber and slide the sample holder onto the stage in the specimen chamber. If you feel too much resistance, the rod may be slightly off center. Adjust the rod position with the black knob on the front of the sample exchange chamber until the sample slides onto the stage.
- Unscrew the rod from the sample mount, fully retract the rod, and lock the rod into place with the latch. Be careful not to push down on the end of the rod while engaging the latch.
- Press **OPEN** button (light will go out).
- Press **CLOSE** button (light will illuminate) and the door between the sample exchange chamber and the specimen chamber will close.
- ### Return to the left hand LCD, and click **OK** and **RESUME EXCHANGE**. Wait until Smart SEM moves the stage from the load position to the viewing position. Click OK to answer the pop ups and close the sample exchange window.

OPERATION:

- If the chamber camera is off, turn it ON.
- Maximize the Smart SEM window.
- Ctrl-G brings up the SEM control panel. Move it to the right hand screen with the mouse.
- Click on the **STAGE** tab of the SEM control panel. Using the left hand joystick, carefully raise the stage (+y) without tilting it (+/-x). Keep one eye on the specimen chamber with the camera, and the other eye on the “tilt” entry on the SEM control panel. Stop when the sample is at a medium distance from the lens.

- Press the **CAMERA** button on the keyboard to turn the camera OFF.
- Click on **EHT** on the bottom of the left hand LCD and click **EHT ON**. Use the **GUN** tab on the SEM control panel to change the accelerating voltage. 5 kV is usually a good starting point, unless the sample is exceedingly thin or has charging problems. The screen should brighten, but the image will probably be out of focus.
- Ctrl-D brings up the “data zone” at the bottom of the image. This will be part of your saved images when it is ON. Ctrl-D toggles this feature off and on.
- Select the **APERTURE** tab on the SEM control panel to choose an aperture. The 30 um aperture is a general purpose aperture and a good place to start. Seven apertures are available ranging in size from 7.5 um to 120 um. You may change the aperture at any time, but adjustments for astigmatism and aperture centering may be necessary to achieve an optimum image.
- Select the **DETECTOR** tab on the SEM control panel and choose the secondary detector.
- Toggle the coarse/fine bar to coarse with the mouse and focus the sample. Increase magnification, toggle to fine, and focus again.
- Check the working distance on the data zone. To adjust, turn the camera on and raise or lower the stage with the joystick, avoiding any unintentional tilt. Then focus again. Repeat until desired working distance is achieved.
- The right hand joystick controls lateral motion (x/y) and rotation (twist). All stage motion may also be controlled from within the **STAGE** tab of the SEM control panel. Find the desired features and manipulate the stage to position the sample.
- The **SECONDARY** detector is usually satisfactory for moderate to long working distances and the entire range of accelerating voltages.
- For superior SE image quality at low accelerating voltages (3 kV or lower) and short working distances (2 to 5 mm), the **IN-LENS** detector is generally preferred. The **IN-LENS** detector may be used **UP TO 20 kV**, but image quality may degrade as working distance increases. Do not use the **IN-LENS** detector above 20 kV; use the **SECONDARY** detector instead.
- The **ESB** is a high-resolution enhanced backscatter and secondary detector that may be useful for working distances of 5 mm or less. **QBSD** is a 15 mm 4-quadrant backscatter detector that must be manually engaged and has not been used yet.

- Choice of detector is mainly determined by image quality, within the limits described above. The **IN-LENS** and **SECONDARY** detectors are the most commonly used.
- With the chosen detector and aperture in place, the accelerating voltage selected, and the feature of interest on the screen, adjust contrast and brightness.
- If there is a large amount of astigmatism present, perform a preliminary correction with the x/y astigmatism controls on the keyboard. Center the aperture by pressing the **WOBBLE** button on the keyboard. You may adjust the amplitude in the **APERTURE** tab of the SEM control panel. Use either the aperture centering knobs on the keyboard or the mouse controls in the SEM control panel. Focus and correct again for astigmatism, using either the keyboard or mouse controls.
- Shift-F2 activates lens clear. Use this if you are unable to correct the astigmatism or have an otherwise unsatisfactory image. If there is hysteresis in the lens, the image will shift and go out of focus. Focus again. Repeat lens clear/focus two or three times if necessary until you can obtain a satisfactory image. If there is still a problem, contact super-user.

SAVE IMAGE:

- When the image is optimized, you may choose to save it. Within the SEM control panel, you can choose from several types of scans (line/frame average, line/frame integration, pixel average) and speeds.
- With averaging, choose a speed and click on **FREEZE** in the SEM control panel when you are ready to save. On the left hand LCD, click on **FILE** and **SAVE IMAGE**. Click on **CHANGE DIRECTORY** then choose drive D and open your image folder. Create a sub-folder if you wish, type in a file name and press **SAVE** or **ENTER**. The micron bar appears on the data zone, but the magnification does not. If you want to save the magnification with your image, append it to your filename (such as “image1_120kx”). In the SEM control panel, click on **UNFREEZE**, then return to **PIXEL AVERAGE** and fast speed (perhaps 3) to return to live image.
- With integration, choose the number of frames and wait until SEM control panel indicates the integration is finished. On the left hand LCD, click on **FILE** and **SAVE IMAGE**. Click on **CHANGE DIRECTORY** then choose drive D and open your image folder. Create a sub-folder if you wish, type in a file name and press **SAVE** or **ENTER**. The micron bar appears on the data zone, but the magnification does not. If you want to save the magnification with your image, append it to your filename (such as “image1_120kx”). In the SEM control panel, click on **UNFREEZE**, then return to **PIXEL AVERAGE** and fast speed (perhaps 3) to return to live image.

- To load a previous image, perhaps in order to make and store a measurement on the image, click on **FILE** and **LOAD IMAGE**. Press Ctrl-A to bring up the annotation menu. Move the menu to the right hand LCD, choose the annotation type, and perform the measurement. To save the annotated image, follow the save image procedure described above.

EXCHANGE / UNLOAD SAMPLE:

- Click on **EHT** on the bottom of the left hand LCD and click **EHT OFF**.
- Press the **CAMERA** button on the keyboard. The interior of the specimen chamber will appear.
- Press the **EXCHANGE** button on the keyboard. A dialog box will pop up. When it says "Ready to Exchange" position yourself in front of the sample exchange assembly.
- Wait for the green **PROCEED** light to illuminate. This means the sample exchange chamber is pumped down.
- Verify that the **PUMP** and **CLOSE** buttons are illuminated.
- Press **CLOSE** button (light will go out).
- Press **OPEN** button (light will illuminate). The door between the sample exchange chamber and the specimen chamber will open.
- Gently release the specimen rod latch.
- Carefully, without applying any bending pressure to the rod, slide the sample exchange rod into the specimen chamber and gently screw the specimen rod into the sample holder. Do not overtighten. Gently slide the sample off the stage, fully retract the rod, and lock the rod into place with the latch. Be careful not to push down on the end of the rod while engaging the latch.
- Press the **OPEN** button (light will go out).
- Press the **CLOSE** button (light will illuminate) and the door between the sample exchange chamber and the specimen chamber will close.
- Press **PUMP** button (light will go out).
- Press **PURGE** button (light will illuminate). You will hear nitrogen flow into the sample exchange chamber and the door will be released.

- Pull the door back and hook the door latch to hold it open.
- Unscrew the rod from the sample holder and remove the sample holder from the mount.
- **You may load another sample at this time if desired.** If loading another sample, slide the sample holder onto one of the two mounts and gently screw the sample exchange rod into the sample holder. Do not overtighten. In any case, now proceed to the next step.
- Release the door latch and gently push the door closed.
- Press **PURGE** button (light will go out).
- Press **PUMP** button (light will illuminate) and the sample exchange chamber will pump down.
- *If another sample has been loaded, return to the top of page 3, and proceed according to the **SAMPLE LOADING** instructions, starting with step *** and continuing through step ###.*
- *If another sample is **NOT** to be loaded, wait for the green **PROCEED** light to illuminate to verify that the sample exchange chamber is pumped down, then continue with the next steps.*
- When finished, verify that all samples have been removed from the specimen and sample exchange chambers, and the **PUMP** and **CLOSE** buttons on the sample exchange chamber are illuminated.
- Return to the left hand LCD, and click **OK** and **RESUME EXCHANGE**. Wait until Smart SEM moves the stage from the load position to the viewing position. Click OK to answer the pop ups and close the sample exchange window.
- Log out of Smart SEM to disable the SEM interface and make it available for the next user. **Note: As the result of a software upgrade, clicking the “X” in the upper right hand corner no longer logs you out, even though it closes SmartSEM.** After logging out, answer “yes” to the pop-up window. This will end your Smart SEM session and allow the next user to log on. Remember that you still must enable and disable the tool in Coral.
- Go into My Computer at the bottom of the left-hand LCD to copy your images from your image file on drive **D:\images\your user ID** to your CD/DVD or flash memory. If you are using a USB connection, please use the hub on the table to connect.